

# IMPACTING CONSTRUCTION THROUGH CONCRETE SCIENCE FOCUSED ON SUSTAINABLE AND ROBUST TRANSLATIONAL RESEARCH

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## 1. PRELUDE

The article focuses on the original contributions made towards the science involved in concrete technology. The purpose of this article is to highlight the scientific contributions made in the field of concrete science at Malaviya National Institute of Technology (MNIT) Jaipur and Indian Institute of Technology (IIT) Indore, with the aim of inspiring the next generation of researchers and engineers.

The research at MNIT Jaipur initially focused on composite structures and later expanded to concrete, with a focus on sustainability and industrial applicability, at both MNIT Jaipur and IIT Indore. The researchers have focused on multiple aspects of concrete science, including alternative materials, microstructural characterisation, product engineering, conventional and emerging tests, development of safety guidelines, and sustainability assessment. The article systematically accounts for studies related to materials used to prepare concrete, fresh state rheology, preparation process and safety. In materials, a special emphasis has been given on supplementary cementitious materials (SCM), followed by new insights on other alternative construction materials. In rheology, the discussion emphasises the advancement towards the understanding of the rheological behaviour of concrete. In the preparation process, the discussion covers different studies related to the mixing and casting of concrete. And, in safety, the article discusses about the coupled deterioration mechanism, and the use of probabilistic models for the development of safety guidelines.

A list of key contributions discussed in the present manuscript is as follows:

- i. Use of characterisation-based assessment for classification of new and alternative SCMs. Extension of the classification-based approach towards optimisation of SCM.
- ii. New and alternative materials covering the use of food waste in bio-cementation, new treatments for construction and demolition (C&D) wastes, and fine bone china ceramic aggregates.
- iii. New functional properties of existing materials, covering the use of geopolymers for colour retention, cow dung as foaming agent and waste tyre rubber concrete for slip and fall preventing pavement.
- iv. Improvement towards the understanding of the rheology of cement through extensive review and experimental analysis for cement paste in a fully broken-down state.
- v. Thermally activated structuration in geopolymer rheology and its demonstration for 3D printed applications.
- vi. Effect of modifying mixing and compaction techniques on the properties of finished concrete.
- vii. Observations related to the synergetic effect of the coupled deterioration mechanism.
- viii. Extension of the Monte Carlo simulation from the risk assessment model to a risk mitigation framework for developing safety guidelines.

## 2. OPTIMISED SELECTION OF SCM USING A CHARACTERISATION-BASED ASSESSMENT

SCMs are mineral powders that are blended with cement to reduce the environmental impact associated with clinker. SCMs take part in multiple stages of cement hydration to improve the resulting microstructure. Based on an extensive literature review, the interaction of SCM in cement hydration can be summarised as a two-stage process<sup>[1]</sup>. In the first stage, the minerals from cement and SCM dissolve in water. The rate of dissolution in water is favoured by the amorphous content, surface area, and availability of favourable minerals like  $\text{Ca}^{2+}$ [1]. In the second stage, the dissolved minerals interact to produce hydrated cement phases. This phase primarily depends on the availability

of dissolved phases and reaction kinetics<sup>[1]</sup>. The hydrated cement phases precipitate on available surfaces, in a phenomenon known as nucleation. Nucleation over a surface hinders its dissolution and acts as a limiter to the first stage of hydration<sup>[1]</sup>. SCMs with higher specific surface area and Ca<sup>2+</sup> content tend to attract precipitating phases and reduce nucleation over unreacted cement phases<sup>[1]</sup>.

### 2.1 Characterisation-based assessment

Higher clinker substitution through SCM has always been advocated for improved sustainability<sup>[2]</sup>. However, an extensive review of the legal framework, availability of raw materials and consumption patterns revealed a major challenge associated with the selection of SCM and optimisation of its dosage<sup>[1,2]</sup>. It is important to note that higher clinker substitution is permitted within the legal framework, but requires additional testing for material safety, which increases the project cost<sup>[2]</sup>. The stakeholders further face the challenge of different types of SCMs with disproportionate geographic availability<sup>[1]</sup>. Furthermore, most available literature classifies SCMs based on the source of their origin. Several studies show that SCMs from a similar source can perform significantly differently in concrete, limiting the replicability of results and stakeholder confidence<sup>[1]</sup>. As an alternative strategy, the article advocates for a characterisation-based assessment approach to select and optimise the dosage of SCMs in concrete.

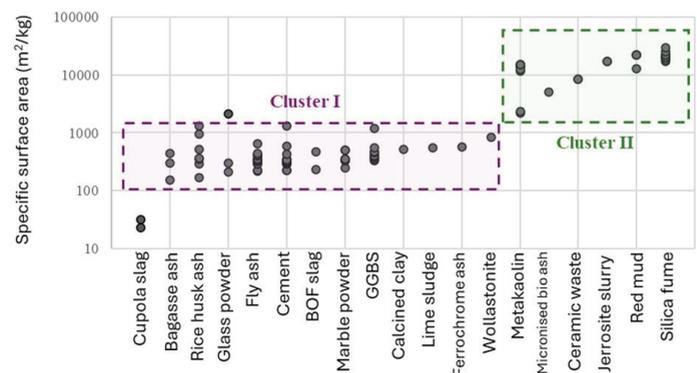
In one of the previous studies, an extensive meta-analysis was carried out to correlate the physical, chemical, and microstructural characteristics of SCMs with their effect on the properties of concrete<sup>[1]</sup>. The study showed that specific surface area and elemental composition have a strong correlation with mechanical, rheological and durability properties of concrete as compared to specific gravity, mineral composition and chemical composition<sup>[1]</sup>. The particle morphology affects the rheological properties of fresh concrete, but does not correlate with other properties<sup>[1]</sup>. Different SCMs were then grouped based on specific surface area and elemental composition, as shown in Figures 1 and 2<sup>[1]</sup>.

The groups demonstrated a strong correlation with the resulting properties of concrete in terms of workability, strength (compressive, flexural, split tensile), and durability (permeability, sulphate attack, chloride ion penetration, carbonation, and wetting-drying cycles)<sup>[1]</sup>. The study shows that, to improve workability, cluster I (similar particle size) with a smooth, spherical morphology should be used, such as fly ash, ground granulated blast furnace slag (GGBS), silica fume, marble powder, and glass powder<sup>[1]</sup>. To improve strength and durability, it is recommended to use cluster II (very fine particles) and materials with high Ca content, i.e., group III (calcareous alumina silicate), group IV (slag) and group V (calcites)<sup>[1]</sup>. It is interesting to note that at high

dosage, cluster II starts to disturb the effective w/c available for hydration, reducing the observed strength improvement<sup>[1]</sup>. At higher dosage, group I (silicate) and group II (alumina silicate) exhibit better sustainability without negatively affecting the strength<sup>[1]</sup>. Furthermore, group I (silicates) are recommended in concrete with higher portlandite availability, like C<sub>3</sub>S cement composite, as they can produce more pozzolanic reactions<sup>[1]</sup>. Higher concentration of soluble aluminium in alumina silicate, calcareous alumina silicate and slag can limit the solubility of silicates and overall pozzolanic activity<sup>[1]</sup>. Group II (alumina silicates) are recommended for concrete with lower portlandite availability, like C<sub>2</sub>S cement composite, for achieving high clinker substitution<sup>[1]</sup>.

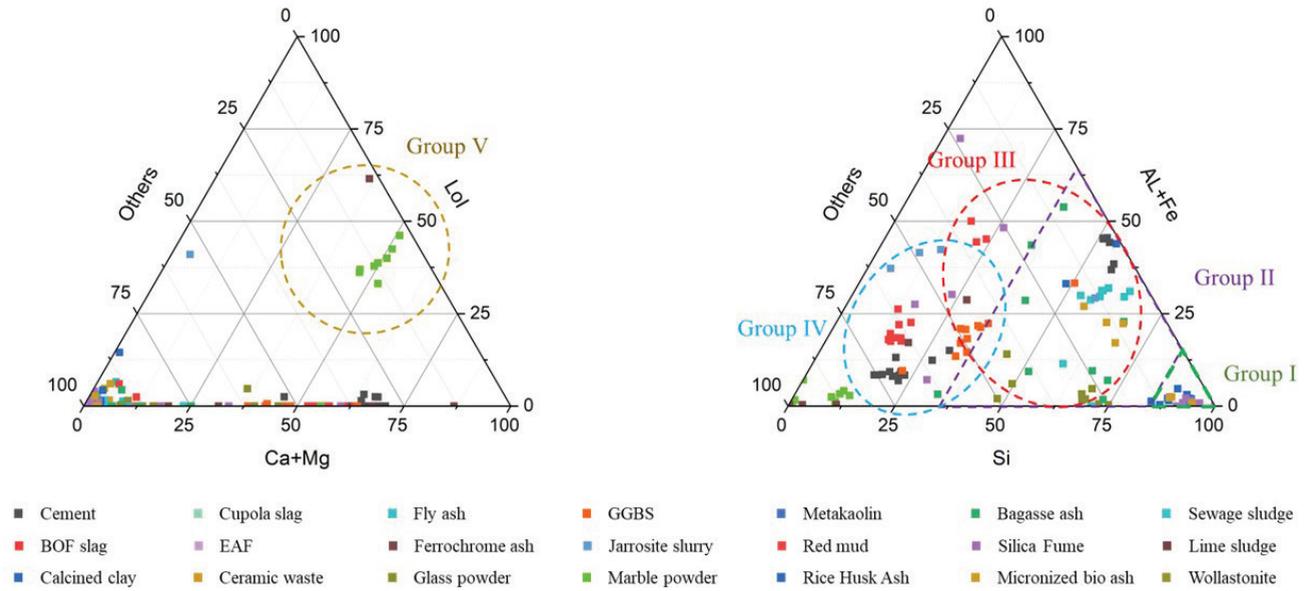
*Key contribution:* The results clearly demonstrate that SCMs with similar specific surface area and elemental composition can be grouped together irrespective of their nature of origin. The study, for the first time, demonstrated the application of grouping across materials from different sources of origin. The classification helps in optimising the use of new and alternative materials as SCMs, based on the desired concrete application.

*Limitations:* Assumptions were taken to account for the limited reporting of characterisation data. In the case of limited reporting, it has been assumed that the missing properties of any given SCM will be within the range reported across other reported studies on the same material. Due to the assumption, the study fails to establish a correlation between material characteristics and the relative performance of different SCMs within the same group of materials. Furthermore, the effect of amorphous content, electrical conductivity, pH and carbon content was reported for very limited SCMs. Further inclusion of these parameters may improve the characterisation-based classification.



Clusters	I	II
Trait	Similar specific surface area <1100 m <sup>2</sup> /kg (typically 200 – 600 m <sup>2</sup> /kg)	Fine particles >1100 m <sup>2</sup> /kg (typically 12000 – 20000 m <sup>2</sup> /kg)

Figure 1: Grouping of SCMs based on specific surface area



Group	Category	Identifying composition	Relevant IS code	SCM
I	Silicate	Si ≥ 85	IS: 15388 (2003) <sup>[3]</sup>	Rice husk ash, Silica fume, Micronized biomass silica,
II	Alumina silicate	(Si+Al+Fe) ≥ 70, Si ≥ 35	IS: 3812-2 (2013) <sup>[4]</sup>	Calcined clay, Ceramic waste, Glass powder, Fly ash, Metakaolin, Bagasse ash,
III	Calcareous alumina silicate	(Si+Al+Fe) ≥ 50, Ca ≥ 10, Si ≥ 25	IS: 3812-2 (2013) <sup>[4]</sup>	Sewage sludge ash, Cupola slag
IV	Slag	CaO+MgO+Al <sub>2</sub> O <sub>3</sub> ≥ SiO <sub>2</sub> , CaO+MgO+1/3.Al <sub>2</sub> O <sub>3</sub> ≥ SiO <sub>2</sub> +2/3.Al <sub>2</sub> O <sub>3</sub>	IS: 12089 (1987) <sup>[5]</sup>	BOF slag, GGBS, Jarosite slurry, EAF
V	Calcites	MgO+CaO+LoI ≥ 50, CaO ≥ 30	-	Marble powder, Lime sludge, Wollastonite

Figure 2: Grouping of SCMs based on elemental oxide composition

## 2.2 Fast search and selection approach

The elemental composition and specific surface area-based grouping can be used to identify a suitable SCM based on the desired concrete application<sup>[1]</sup>. The grouping system, while being able to identify potentially suitable SCMs, did not indicate optimum substitution or effect on overall sustainability. In another study, the application of the characterisation-based approach was further extended by integrating a probability-based mathematical model<sup>[6]</sup>.

The mathematical model uses the probability of success  $[r(\phi)]$  for a given degree of substitution  $(\phi)$ , as expressed in Equation 1<sup>[6]</sup>. The expression is derived from the previous trials on optimum composition and elemental composition, as shown in Equation 2<sup>[6]</sup>. The probability of success can then be correlated with potential savings after accounting for costs related to additional testing, as shown in Equation 3-4<sup>[6]</sup>. Here, Equation 3 serves as the basis for a fast search and selection approach, which can help the stakeholders to understand how long to look for an alternative SCM and when to stop looking.

$$r(\phi) = \sum p(\phi_p) \in \phi_p \geq \phi \tag{1}$$

$$p(\phi_p) = \frac{1}{\sum_k \left( \frac{\sum (\text{oxide \% of SCM } i - \text{oxide \% of SCM } k)^2}{1} \right)} \tag{2}$$

$$\text{Net savings} = r_n(\phi) \times k_T \times V \times (C_{cem}(\phi - \phi_0) + (C_0 \times \phi_0 - C_i \times \phi)) - \sum_{i=1}^n \sum_{j=1}^m (C_{testj}) \tag{3}$$

$$r_n(\phi) = 1 - \prod_{i=1}^n (1 - r(\phi)_i) \tag{4}$$

Here,  $\phi$ ,  $\phi_p$ ,  $\phi_0$ ,  $p(\phi_p)$ ,  $r_n(\phi)$ ,  $k_T$ ,  $V$ ,  $C_{cem}$ ,  $C_0$ ,  $C_i$ ,  $C_{testj}$ ,  $n$ ,  $m$ , and  $k$ , represent the degree of substitution for which savings are being estimated, and the optimum substitution level based on previous trials for the data set  $k$ , current level of SCM usage, probability of optimum substitution being  $\phi_p$ , the probability of at least one successful exploration across  $n$  trials, a factor for discounted present value across the time duration  $T$ , volume of cement to be used, cost of cement, cost of presently used SCM,

and landed cost of SCM used in trial  $i$ , cost of test  $j$ , number of trials, number of tests, and data set  $k$  based on previous trials, respectively.

**Key contribution:** The mathematical equation demonstrates the principles of macroeconomics in civil engineering applications by introducing probability functions. The approach presents a novel framework that, when fully evolved, can quantify the amount one can spend on research while ensuring profit. This will effectively encourage stakeholders to explore alternative materials based on a quantified matrix.

**Limitation:** The mathematical equation was validated over a small dataset, *i.e.*, chemical composition of 30 different fly ashes, for the maximum replacement level without any strength loss. Full-scale validation across different SCMs for different material characteristics has not been completed. Furthermore, in the limited results, the model predicted a conservative success rate, *i.e.*, up to 8 % lower theoretical replacement level than the experimental optimum. In the present format, the SCMs will be suggested at lower replacement levels, and further experiments will be needed to increase SCM utilisation to optimum replacement levels.

### 2.3 New observations related to SCM characterisation

Previous studies also explored the application of different characterisation techniques on various SCMs<sup>[7,8]</sup>. Some of the observations, with potential for improving further understanding of the SCMs, have been summarised as follows:

- i Irregularly shaped particles exhibit higher pozzolanic reactivity than spherical particles when observing different coal-based fly ashes<sup>[8]</sup>.
- ii Raman spectroscopy can detect and quantify unburnt carbon in different coal-based fly ashes, making detection faster than loss on ignition testing.
- iii Raman micrograph can identify particle distribution and confirm the degree of blending between different siliceous and calcareous powders<sup>[7]</sup>.

It should be noted that these observations are based on a few SCMs. As a result, these may be true for specific SCMs and may not be a generalised observation. However, these observations can improve the characterisation of SCMs and need further exploration in future research.

## 3. INSIGHTS ON NEW MATERIALS AND ALTERNATIVE APPLICATIONS

### 3.1 Food waste as nutrient media for bio-cementation

Food waste and cement manufacturing are two major contributors to global carbon emissions<sup>[9]</sup>. Literature shows that sequestering CO<sub>2</sub> in concrete can lower the carbon footprint and improve the properties of concrete<sup>[9]</sup>. The conventional strategies for carbon sequestration focus on captured CO<sub>2</sub>. In a previous study, CO<sub>2</sub> released through the controlled decomposition of food waste was explored as an alternate route for carbon sequestration in concrete<sup>[9]</sup>. Here, food waste, along with compatible non-pathogenic bacteria, is added to concrete. The bacteria will feed upon the food waste to release CO<sub>2</sub>, which is simultaneously sequestered by portlandite, as shown in Figure 3<sup>[9]</sup>. The alternate route eliminates the need for trapping CO<sub>2</sub> while replacing the commercially used nutrient media during bio-cementation with upcycled food waste<sup>[9]</sup>.

The study explored two major pathways for upcycling food waste as nutrient media, *i.e.*, powder form and pulp form<sup>[9]</sup>. The study showed high bacterial growth in powder-based nutrient media for *E. Coli DH-5α*. The solution was added in a mortar by mixing water and bacteria immobilised on waste tyre rubber fibre<sup>[9]</sup>. The resulting microstructure showed that bacteria addition resulted in a denser cement matrix and improved interfacial transition zone (ITZ) around rubber fibres, as shown in Figure 4. The resulting mortar increased the strength by over 80 % for mortar prepared with up to 2 % rubber. This is significantly higher than previously reported works on bacterial mortars, with a maximum strength gain of about 60 % across all observed literature<sup>[9]</sup>. The bacterial rubberised mortars also exhibited self-healing ability<sup>[9]</sup>. It is hypothesised that the higher strength gain may be a result of the smaller size of *E. Coli DH-5α*. A smaller bacterial size will also be able to occupy smaller pores and occupy them through calcite precipitation, resulting in a denser microstructure.

The study also demonstrated that the bacteria immobilised on the surface of the rubber can strengthen the ITZ<sup>[9]</sup>. The process of bacteria immobilisation can be extended to other wastes, like plastic, for improved waste upcycling.

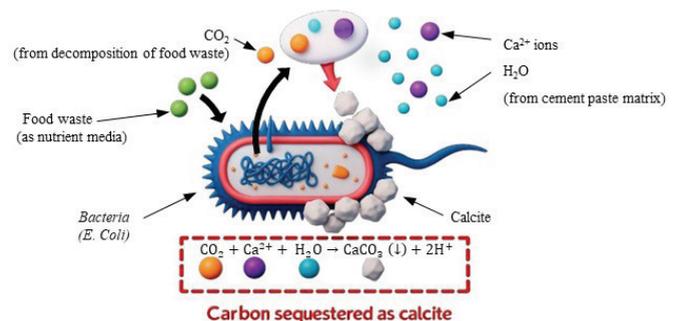
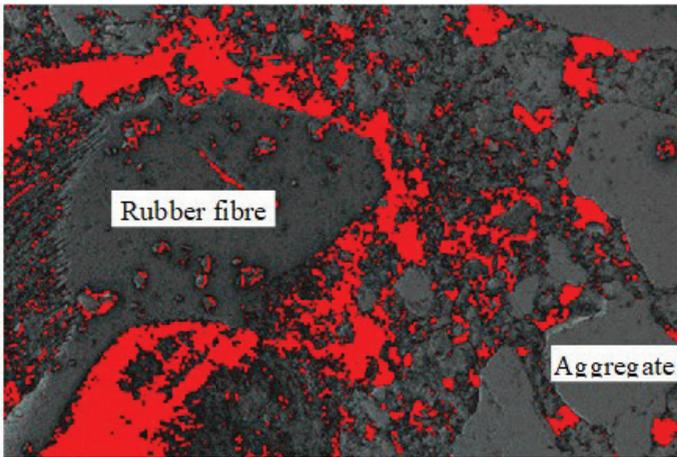
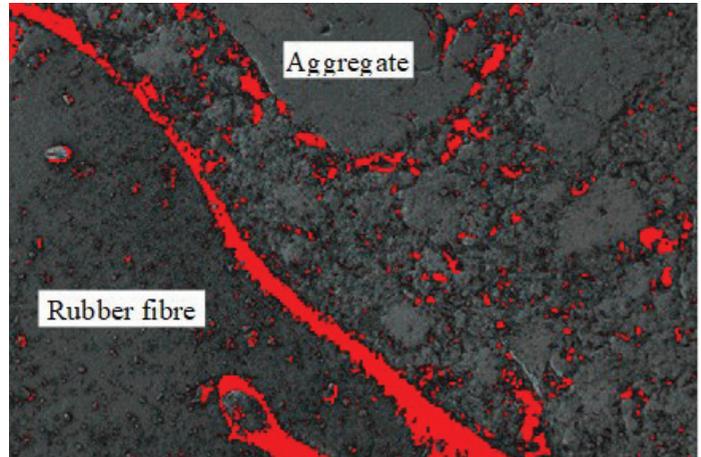


Figure 3: Carbon sequestration using food waste for bio-cementation



(a) Normal mortar 18.1 % ITZ and matrix pores (in red)



(b) Bacterial mortar 8.0 % ITZ and matrix pores (in red)

Figure 4: Effect of food waste and bacteria on the microstructure of mortar<sup>[9]</sup>

**Key contributions:** The study demonstrates that the use of food waste can eliminate the need for synthetic nutrient media, one of the highest cost contributors, and make bio-cementation more cost-effective. The study also observes that the bacterium *E. Coli DH-5α* favours strength enhancement over crack-filling ability. This can inspire exploration of different bacteria as specialised bio-admixture, extending beyond self-healing applications.

**Limitations:** The study has been limited to mortars and a specific set of food wastes. Further research is ongoing towards identifying a suitable non-pathogenic bacterium that can work with a wide range of food wastes. Furthermore, the hypothesis for the effect of bacteria size on strength is based on an observed inverse correlation between the strength and size of bacteria across multiple literature. Further investigation in this direction is needed to validate the hypothesis.

### 3.2 Treatment of C&D wastes

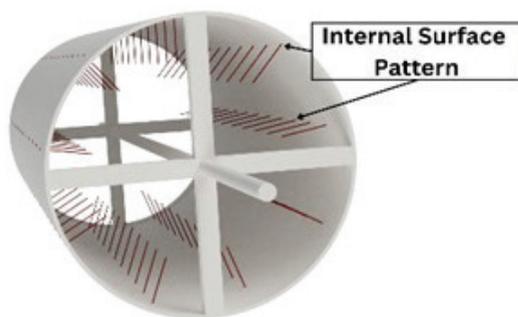
The author has explored various strategies for treatment and upcycling C&D wastes. This includes two novel recycling pathways, *i.e.* cryogenic treatment and surface-modified abrasion, and an improvement in the existing thermo-

mechanical recycling. In addition to this, previous studies have been conducted on a combination of different treatments, waste-based slurry-fibre coating, and different surface treatments. However, the same has not been reported due lack of technical or commercial viability.

Cryogenic treatment uses the freeze and thaw mechanism for weakening of adhered mortar, followed by mechanical scrubbing for removing the weakened adhered mortar<sup>[10]</sup>. The process is slow and yields a small improvement in aggregate characteristics. The treatment establishes that deteriorated C&D waste in cold regions can be upcycled by using abrasive cleaning, without requiring additional weakening of adhered mortar.

In another study, the conventional rotatory abrasion was improved by introducing irregularities on the otherwise smooth drum surface (Figure 5a)<sup>[11]</sup>. The resulting surface irregularities provided additional abrasive action, lower adhered mortar content and lesser energy consumption as compared to material treated in the Los Angeles abrasion machine.

Thermo-mechanical treatment is particularly suited for the treatment of mixed recycled aggregates prepared from



(a) Surface irregularities



(b) Rotatory device

Figure 5: Prototype for the surface-modified abrasion process

unsorted C&D wastes, which is one of the challenges for C&D waste management in India. A previous study shows that thermal exposure of 700 °C produces the optimum aggregate characteristics, by weakening of adhered mortar, combustion of organic impurities and sintering of clay materials<sup>[12]</sup>. The weakened adhered mortar is then removed by abrasion and sieve segregation. The resulting aggregate satisfies all codal provisions for high-quality aggregates, as stipulated in IS: 383 (2016)<sup>[12]</sup>. At 100 % substitution of natural aggregates, the treated aggregate showed over 90 % mechanical strength<sup>[9]</sup>. The treatment also results in the generation of recycled fine aggregates and recycled fine powder, which show higher strength than natural river sand and fly ash in mortars.

*Key contributions:* Among the different studies, the introduction of surface irregularities during abrasion is considered a novel contribution with potential for impact. The results prompt a new direction for improving abrasive cleaning of adhered mortar, which is widely used in the observed literature. In the case of thermo-mechanical treatment, the previous study demonstrated the suitability and industrial scalability of the solution for mixed recycled aggregates.

*Limitations:* The results on surface irregularities are based on 20 mm recycled concrete aggregates from the same batch. The efficiency of the modification may be influenced by arrangement and type of irregularities, rotation speed and duration, grade of parent concrete, degree of adhered mortar, and presence of particles other than concrete. Further investigations in this direction can show the overall viability of smooth and irregular surfaced abrasive treatment.

### 3.3 Alternative materials with functional applications

#### 3.3.1 Naturally coloured geopolymer

Dimensional stone wastes, like marble slurry, retain the colour of their parent rocks. Preparation of geopolymer using GGBS and

dimensional stone wastes as precursors preserves the natural colour of the stone in the resulting geopolymer<sup>[13]</sup>. Results show that a higher ratio of dimensional stone waste in the precursor increases the colour intensity, but reduces strength. The composition can be optimised to produce a naturally coloured geopolymer<sup>[13]</sup>. The naturally coloured geopolymer has been used to produce a coloured-layer in bi-layered bricks and other building materials<sup>[13]</sup>.

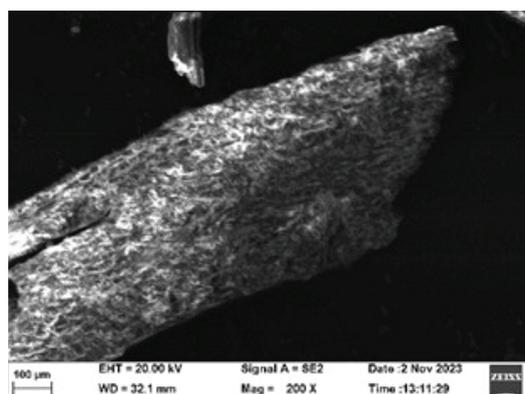
*Key contributions:* The study demonstrated the colour-retaining properties of geopolymer. Multiple repetition with different stone wastes, precursor and activator concentrations confirms the same.

*Limitations:* The developed geopolymer, in many cases, shows a significant reduction in strength, indicating reduced sustainability. Furthermore, both colour and strength significantly depend on the precursor, making the solution susceptible to raw material availability. Further investigations are needed to develop a geopolymer design mix with consistent strength, colour and lower susceptibility to precursor changes.

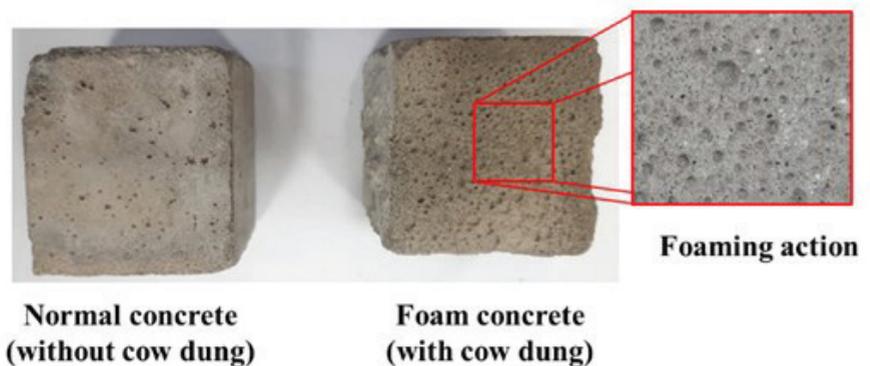
#### 3.3.2 Cow dung as foaming agent

Cow dung particles show a porous, irregular surface with a hollow tube-like structure (Figure 6a), resulting in high moisture retention. When added to concrete with excess water, the particles retain the moisture without disturbing the stability and flowability of the fresh mix. On hardening, the excess water is taken up by the concrete, creating voids and imparting internal curing. The voids are uniformly distributed throughout the concrete matrix and recreate an action similar to a foaming or aerating agent (Figure 6b). Furthermore, the internal curing action provides better strength than foam concrete at small dosages of cow dung powder.

*Key contributions:* The study demonstrated a first-of-its-kind foaming agent based on moisture pockets, as compared to previously reported foaming agents. The results were validated



(a) Microstructure of cow dung particles



(b) Effect of cow dung on concrete

Figure 6: High moisture retention of cow dung and its application for foaming action

across different dosages, excess water, particle sizes, animal dung, processing techniques, freshness of dung, and age of the prepared powder. The consistency of results across repeated testing helped demonstrate the application of cow dung as a foaming agent.

*Limitations:* Due to high moisture content, the foaming agent failed to demonstrate densities below  $1200 \text{ kg/m}^3$  or compatibility with steam curing. Furthermore, the developed foaming also alters the strength development in the prepared mixes, limiting the scope of its application. At higher dosages, early age strength development is delayed. While it is speculated to be a result of  $\text{Ca}^{2+}$  distribution due to a higher effective  $w/c$  ratio, the specific mechanism is under further investigation. Due to lower early age strength, for mortars with a density below  $1600 \text{ kg/m}^3$  demoulding period increased from 16 h to up to 40 h. Cow dung has shown an additional property of internal curing with up to 90 % strength in the absence of external curing, as opposed to 60 % strength in control samples. Further investigations are needed to optimise the application of cow dung as an internal curing agent.

### 3.3.3 Waste tyre rubber and its specialised application

Waste tyre rubber has been upcycled in concrete in the form of crumbs, chips, powder and fibres<sup>[14]</sup>. The addition of tyre rubber is typically detrimental to the compressive strength of the concrete and not recommended for most applications<sup>[11]</sup>. Interestingly, the addition of waste tyre rubber in fibre form improves the flexural strength, energy absorption capacity and surface friction of concrete, suitable for pavement applications<sup>[15-18]</sup>. For different compositions of rubber fibre-added concrete, as compared to control, energy absorption can be increased by up to 30 % in terms of drop impact, energy absorbed till the first crack can be increased by up to five times, and abrasive wear can be reduced by about 45 %, implying better material durability<sup>[15-18]</sup>. Furthermore, the friction coefficient can be increased by 0.15, resulting in reduced slip and fall probability. At the same time, energy absorbed results in reduced rebound energy, which can reduce the fall-related injuries<sup>[18]</sup>. The improvement in energy absorption and abrasive resistance can also be observed for rubberised ash, obtained after pyrolysis of tyre rubber<sup>[19]</sup>. However, the efficacy of rubber ash is lower than the fibres<sup>[18,19]</sup>.

*Key contributions:* Extensive investigations were conducted to identify viable applications for waste tyre rubber. While some of the scientific insights have been previously reported, studies related to friction and the proposed anti-slip pavement applications are novel.

*Limitations:* The use of rubber also reduces the elasticity of the concrete, which can affect walking comfort and increase the

fall probability. Further investigation in this direction is needed to evaluate walking comfort with the reduced elasticity or to engineer pavement with similar elasticity.

### 3.3.4 Fine bone china ceramic aggregates

Fine bone china is a high-strength ceramic with high Ca (>20 %) content<sup>[20]</sup>. In general, powdered wastes with high Ca content show a good pozzolanic reactivity. However, fine bone china is one observed exception where the waste performs better as an aggregate than SCM. As an SCM, fine bone china ceramic powder can be used as a 20 % substitute for cement. At higher dosage, the high volume of  $\text{P}_2\text{O}_5$  (>10 %) starts affecting the pozzolanic activity. As a fine aggregate, fine bone china can replace up to 60 % of natural sand without showing any detrimental effect on the properties of concrete<sup>[20]</sup>. The fine bone china aggregate supports nucleation, or C-S-H gel deposition, around the ITZ and improves the microstructure of concrete, as shown in Figure 7<sup>[21]</sup>.

The resulting concrete shows better strength (compressive, flexural, split tensile), residual performance after fire exposure, impact resistance, sulphate resistance, chloride resistance, abrasion resistance and acid resistance<sup>[20,22-27]</sup>. Although the strength improvement can be observed up to 100 % replacement, more than 60 % is not recommended due to reduced workability<sup>[20]</sup>. Overall, the fine bone china ceramic aggregate can be used as a partial substitute for sand to develop high-performance concrete.

*Key contributions:* Extensive investigations were conducted to demonstrate the suitability of fine bone china ceramic aggregates as a sustainable replacement for natural river sand. While new scientific insights were not observed, the studies validated the role of Ca towards nucleation and microstructural improvement.

*Limitations:* The observed improvements in the microstructure are attributed to the presence of higher Ca content, based on an extensive literature review. No suitable experiment was identified to validate whether any other factor played a contributing role towards the microstructural improvement.

## 4. RHEOLOGY

The understanding of the rheology of cement composites is derived from observations on non-hydrating colloids and suspensions. The hydration of cement affected properties over time, making it difficult to separate the effect of different factors on the rheological properties at a given point in time<sup>[28]</sup>. Often, the rheology of cement was reported as a contradictory phenomenon across different studies<sup>[28]</sup>. This limited a generalised understanding of rheology and its application for advanced applications like 3D printing. The gap in fundamental

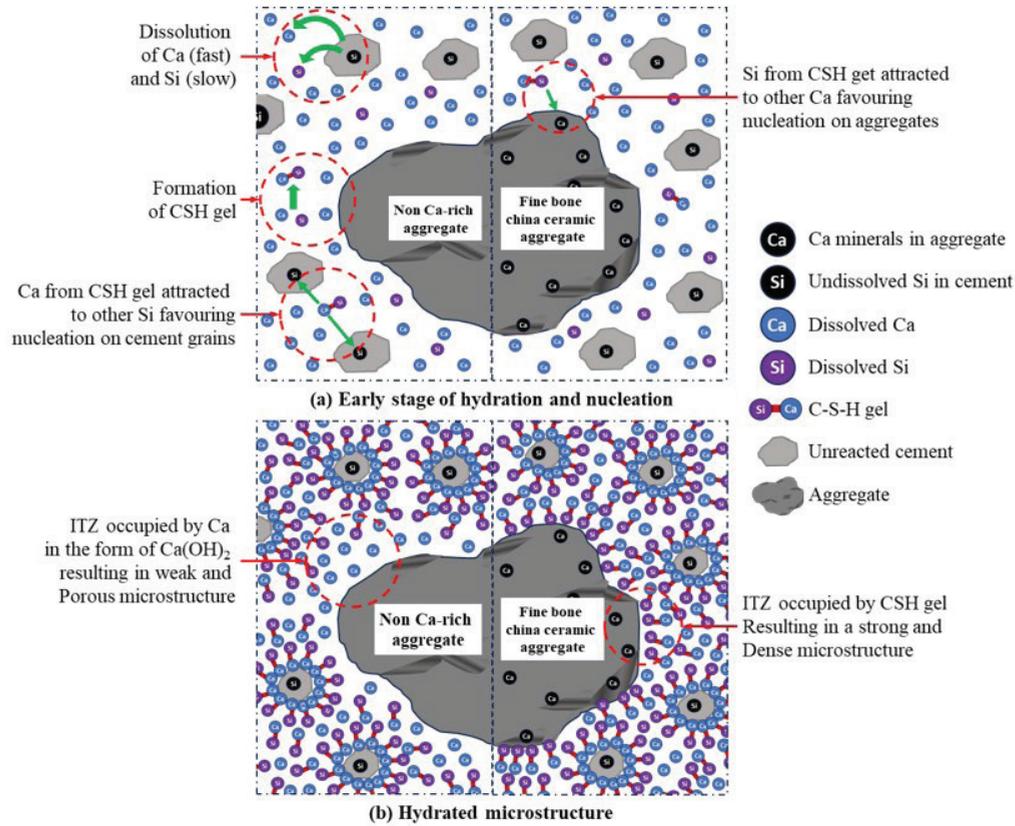


Figure 7: ITZ strengthening in fine bone china ceramic aggregate

understanding of rheology is systematically addressed by understanding the effect of different factors to develop a generalised mathematical expression to describe the rheology of cement composites.

### 4.1 Factors affecting rheology

An extensive literature review was carried out to understand factors influencing the rheology<sup>[28]</sup>. The study focused on observing contradictory phenomena, their study parameters and experimental protocol to develop a deeper understanding towards the change in rheological behaviour<sup>[28]</sup>. The current discussion will focus on the factors that have been reported to exhibit a shift in the rheological behaviour across different studies<sup>[28]</sup>. A simplified explanation describing how the factor is influencing the rheology is also given. Further investigations will be needed to validate the observations and develop corresponding mathematical expressions.

#### 4.1.1 External factors

External factors represent the changes resulting from the environment or the applied shear rate at different times (or shear history) during the casting process. In this regard, temperature and shear history are the primary factors influencing the rheological properties<sup>[28]</sup>.

**Temperature:** The apparent viscosity of the mix increases with temperature at a low  $w/c$  ratio, but decreases at a high  $w/c$  ratio<sup>[28]</sup>. The changes at low  $w/c$  ratio are widely reported in the literature and primarily attributed to water loss from hydration and evaporation<sup>[28]</sup>. At high  $w/c$  ratio ( $\geq 1.2$ ), the behaviour of the cement mix shifts from a viscoelastic (paste) to a Newtonian fluid (suspension)<sup>[28]</sup>. In Newtonian fluids, the properties of the fluid dominate the rheological behaviour<sup>[28]</sup>. On increasing the temperature viscosity of water reduces, which can explain the overall reduction in apparent viscosity at high  $w/c$  ratio<sup>[28]</sup>. Furthermore, at a high  $w/c$  ratio, loss in water due to evaporation and hydration will be insignificant, and will have a limited impact on the overall rheology<sup>[28]</sup>. The overall effect of temperature at different  $w/c$  ratios is shown in Figure 8.

**Shear history:** Shear history represents the shear rate,  $\dot{\gamma}$ , experienced by the mix at different time intervals,  $t$ . Literature suggests that the apparent viscosity of the mix changes with structural build-up due to hydration,  $A(t)$ , and structural breakdown due to the applied shear rate,  $B(\dot{\gamma}, t)$ <sup>[28]</sup>. Shear history is used to determine the net structural buildup remaining in the mix and, accordingly, the rheological properties at a given time, as shown in Equations 5 and 6.

$$\tau_t = \tau_0 + \mu \times \dot{\gamma} + |A(t) - B(\dot{\gamma}, t)| \tag{5}$$

$$\tau_t = \tau_0 + \mu \times \dot{\gamma} + A(t) \times (1 - B(\dot{\gamma}, t)) \tag{6}$$

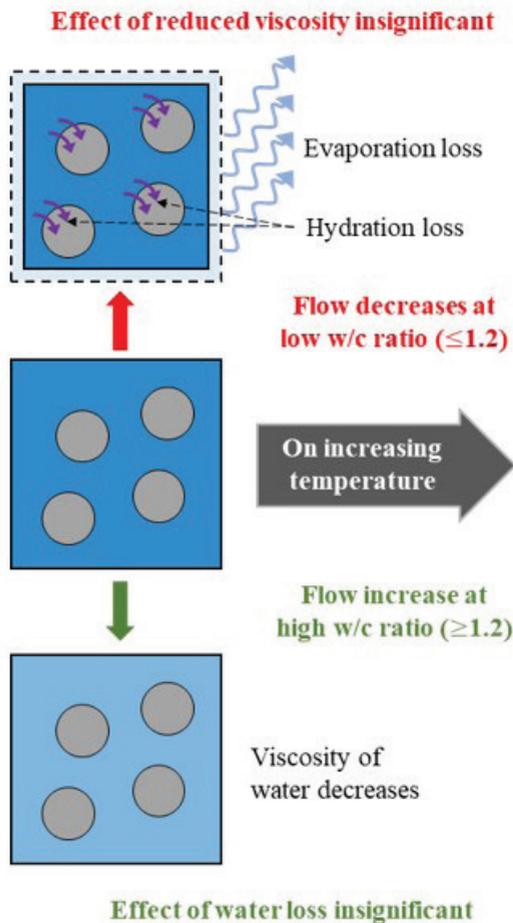


Figure 8: Dominant effect of temperature on rheology at different w/c ratios

In theory, the changes appear to be reversible if structural breakdown exceeds structural buildup. However, the cement mix also experiences irreversible changes due to hydration and resulting microstructural changes<sup>[28]</sup>. An alternate mathematical expression is proposed as Equation 7 to account for irreversible changes<sup>[29]</sup>. The expression consists of two parts,  $\tau_0 + \mu(\alpha) \times \gamma + \tau_1(\alpha)$ , describing the behaviour in a fully broken-down state of cement, and net structural buildup based on shear history,  $\int A(\alpha, \gamma) \times (1 - B(\gamma, t)) \times dt$ . The fully broken-down state uses the degree of hydration,  $\alpha$ , to account for irreversible changes in viscosity,  $\mu(\alpha)$ , and yield stress,  $\tau_1(\alpha)$ . An extended discussion on the development of this expression is given in Section 4.2.

$$\tau_t = \tau_0 + \mu(\alpha) \times \gamma + \tau_1(\alpha) + \int A(\alpha, \gamma) \times (1 - B(\gamma, t)) \times dt \quad (7)$$

Here,  $\tau_t$  represents shear stress at any given time  $t$  under the applied shear rate  $\gamma$ .  $A(\alpha, \gamma)$  and  $B(\gamma, t)$  represents the function of reversible structural buildup and breakdown, respectively.  $A(\alpha, \gamma)$  uses different mathematical expressions based on the stage of hydration. Whereas,  $B(\gamma, t)$  follows exponential decay functions to represent decay and inertial lag.

#### 4.1.2 Internal factors

Internal factors correspond to the constituents of cement paste and their interactions. Among them, several factors, like w/c ratio, aggregate content, aggregate, and admixtures, are well explained in the literature<sup>[28]</sup>. One new observation is regarding the fibre dosage. In rubber fibre-added mortars, after a critical volume, the relative motion of fibres causes greater interaction with other fibres, as compared to aggregates present in the mix<sup>[30]</sup>. This increased interaction results in mechanical interlocking, significantly increasing the static yield strength<sup>[30]</sup>. In a conventional rheological test, one-directional shearing is applied; on rearrangement, the interaction reduces significantly, and a sudden increase in shear thinning can be observed<sup>[30]</sup>. The mechanical interaction can also result in cluster formation or balling effect and affect the homogeneous distribution of fibre during mixing (Figure 9). Further investigation in this direction is needed to determine whether the effect is limited to stiff fibres or extends to flexible fibres.

*Key contributions:* The extensive review identified several experimental factors affecting the rheology of cement composites, which have been reported to exhibit contradictory phenomena. The review shows that the different trends occurred at different experimental ranges, which were reported as contradictory phenomena in the available literature. The review further draws parallels from cement hydration and the rheology of non-cementitious pastes to explain the difference in trends at different experimental ranges.

*Limitations:* The study is based on an extensive review and only theoretically explains the changes in rheological behaviour. Further studies are needed to validate the theoretical explanation through controlled experimental protocols.

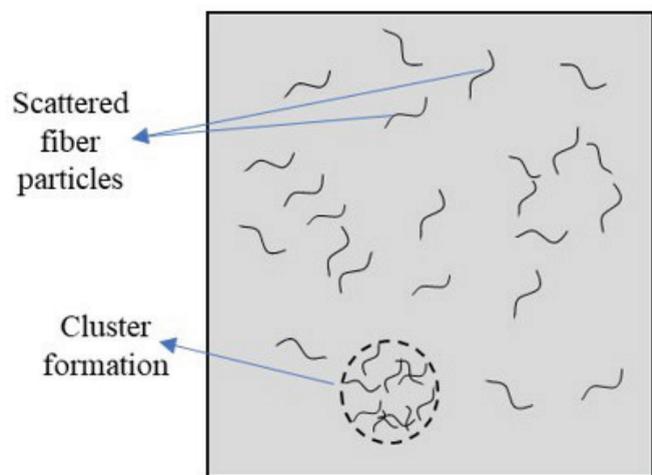


Figure 9: Cluster formation at high fibre dosage

## 4.2 Alternative rheological models and testing methodology

A new test protocol has been designed to understand the irreversible changes by bringing the cement paste to a fully broken-down state, termed 'distinct shear rate protocol'<sup>[31]</sup>. In this protocol, Figure 10, a constant high shear rate is applied to ensure complete structural breakdown<sup>[31]</sup>. The applied shear rate is lowered to a specified value  $\gamma_i$ , at different time intervals  $t_j$ <sup>[31]</sup>. The test is repeated for different values of shear rate at the same time intervals<sup>[31]</sup>. The shear stress vs shear rate at any given time generates the rheological behaviour in a fully destroyed state, Figure 11<sup>[31]</sup>.

The distinct shear rate protocol was applied to understand the rheological behaviour of cement pastes at  $w/c$  ratios of 0.40, 0.45, 0.50 and 0.55 at shear rates of  $0.001 \text{ s}^{-1}$ ,  $0.01 \text{ s}^{-1}$ ,  $0.1 \text{ s}^{-1}$ ,  $1 \text{ s}^{-1}$  and  $10 \text{ s}^{-1}$ <sup>[31]</sup>. The results have demonstrated that the testing protocol can differentiate between the reversible and irreversible changes<sup>[31]</sup>. Further investigations are underway for the cement hydration duration of 10 to 40 min,  $w/c$  ratios of 0.40-0.55, and temperature 15-45 °C, to develop a generalised mathematical model based on the reversible and irreversible rheological changes.

**Key contributions:** The study proposed and demonstrated the

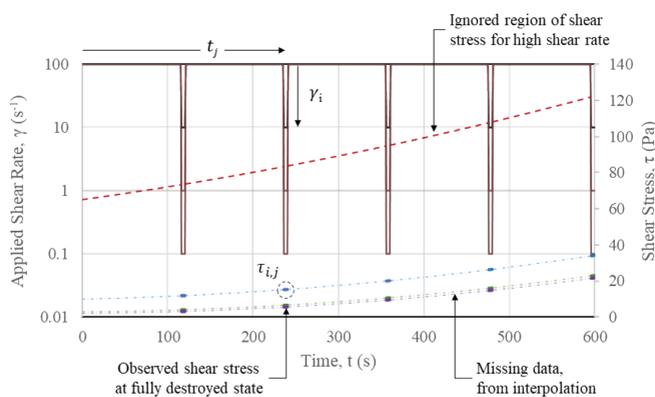


Figure 10: Study of the fully destroyed state using the distinct shear rate test

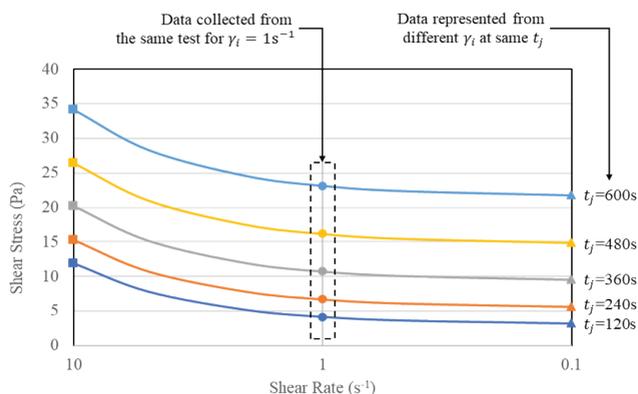


Figure 11: Rheological behaviour in the fully destroyed state

distinct shear rate protocol, which can be applied to study the rheological behaviour of cement paste in a fully broken-down state. The protocol removes reversible structuration and studies the irreversible changes. The study presents the basis towards a better understanding of cement rheology by separately accounting for reversible and irreversible changes.

**Limitations:** The results observed in the proposed distinct shear rate protocol can yield a higher shear resistance than the actual shear resistance of the fully broken-down state due to four factors, which can not be isolated due to experimental limitations. Among them, shear lag (dominates at the start of the observation period) and structuration at reduced shear rate (dominates at the end of the observation period) provide slightly higher shear resistance for the observation period. While boundary effect (dominates at lower  $w/c$  ratio) and segregation (dominates at higher  $w/c$  ratio) provide incremental changes in shear resistance over the test duration. The previous work shows that the effects of these four factors provide a small contribution to the overall shear resistance; hence, they are excluded during the mathematical modeling of the fully broken-down state.

## 4.3 Active rheological control using thermal regulation

In a separate study on heat-cured geopolymers, a constant shear rate of  $1 \text{ s}^{-1}$  was applied over different cement and geopolymeric pastes, at a constant temperature of 20 °C<sup>[32]</sup>. It was observed that the shear stress in GGBS-based geopolymers showed small changes over a long period of time ( $0.014 \text{ Pa/s}$ ) (Figure 12)<sup>[32]</sup>. On application of heat, at a constant temperature of 60 °C, the shear stress suddenly increases at a high buildup rate of  $54.6 \text{ Pa/s}$ <sup>[32]</sup>. Furthermore, a similar build-up is observed for delayed heat application, demonstrating a temperature-dependent and time-independent phenomenon (Figure 12)<sup>[32]</sup>. The results were used to demonstrate a laser-based 3D printing, as shown in Figure 13b<sup>[32]</sup>.

**Key contributions:** The study presents a first proof of concept for a laser-based 3D printing system.

**Limitations:** The process is at a very early stage and needs optimisation in terms of heat delivery and layer stacking. The

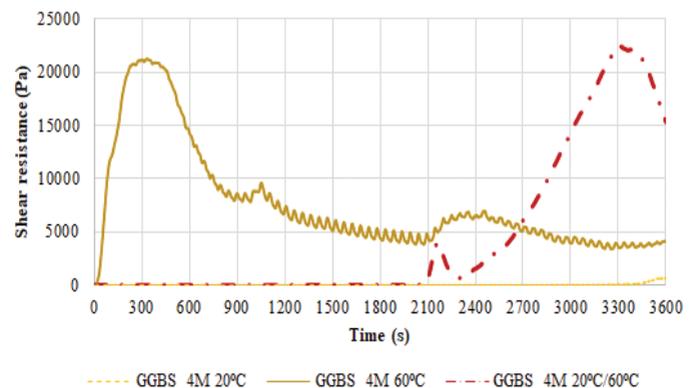
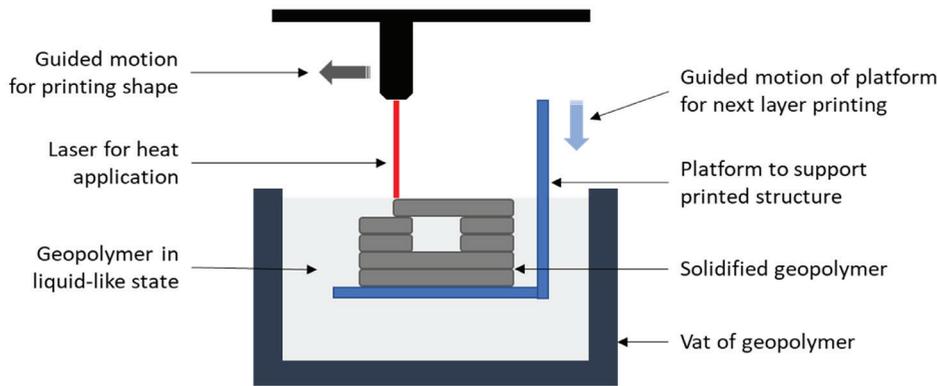


Figure 12: Structural buildup in geopolymer due to a change in temperature



(a) Laser-based 3D printing representative diagram

(b) Prepared sample

Figure 13: Laser-based 3D printing using a heat-cured geopolymer

previous trials showed that during longer heat exposure, the depth of the printed layer increases marginally due to poor heat conductivity. Higher exposure temperature results in shrinkage cracks. In the previous trials, stable layers could be printed up to 3 mm in thickness. Furthermore, in a separate experiment to study the interlayer bonding, the sample was coated with a second layer of geopolymer and heated. It was observed that a small coating thickness caused shrinkage cracks, while a large coating thickness resulted in poor interlayer bonding. Further studies are needed to figure out a method of controlling layer thickness.

### 5. IMPROVING THE SCIENCE OF CONCRETE MIXING AND COMPACTION

In addition to materials, the production process has a significant impact on the properties of building products. Several investigations have been carried out to improve the product quality through the preparation process. Some notable observations have been included in the manuscript.

#### Optimum mixing sequence for low-moisture mixes:

Compression-based fly ash bricks are prepared using low-moisture mixes. Two different mixing methods were explored at different water-binder ratios to compare the effect of mixing sequence on the quality of bricks<sup>[33]</sup>. First mixing sequence used dry blending of solids, followed by water mixing (Figure 14)<sup>[33]</sup>. The second mixing sequence used the preparation of the binder slurry and coating it over the aggregates (Figure 14)<sup>[33]</sup>. Results showed that dry blending followed by water addition produced better quality bricks, in terms of strength, density, water absorption, impact energy and drying shrinkage<sup>[33]</sup>. Here, the poor strength and compaction in the second mixing method suggest that the slurry failed to distribute within the mix due to reduced flowability resulting from low moisture. Therefore, for the adopted mix, the first mixing method is recommended for brick manufacturing, which is the typically adopted industrial method.

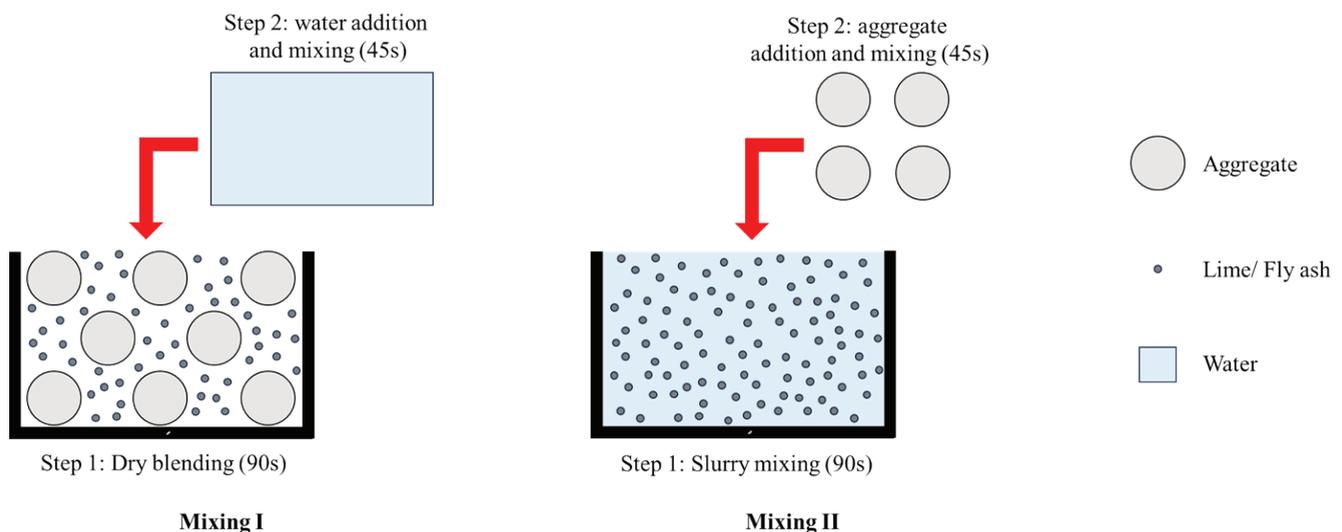
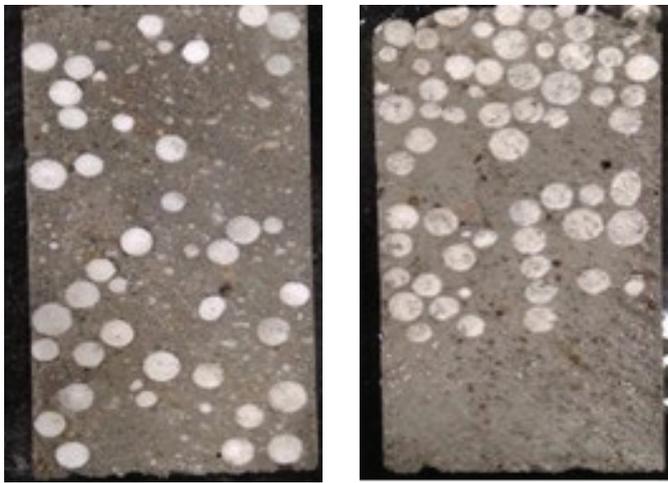
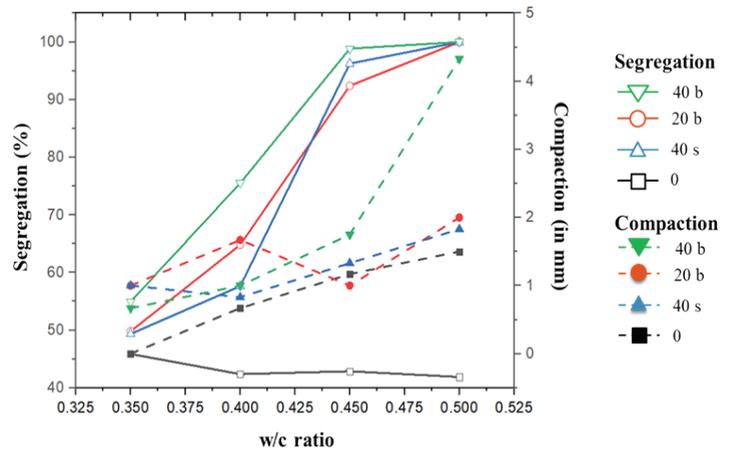


Figure 14: Different mixing sequences for the preparation of low-moisture fly ash bricks



(a) Unsegregated sample

(b) Segregated sample



(c) Comparison between the degree of segregation and compaction

Figure 15: Effect of vibration on the segregation of lightweight aggregates

**Segregation control for lightweight aggregates:** During vibration, lightweight aggregates segregate to the top surface, resulting in a heterogeneous mix (Figure 15b). An expanded polystyrene (EPS) based lightweight coarse aggregate was used to prepare different mix designs and subjected to a simulated vibration-based compaction<sup>[34]</sup>. Three different protocols were used to simulate vibration, representing similar maximum amplitude, similar compaction energy, and similar compaction duration<sup>[34]</sup>. Results showed that segregation primarily depends on maximum amplitude and rheological properties of the mix, rather than the duration of compaction (Figure 15c)<sup>[34]</sup>. Therefore, lightweight aggregates can be compacted for a long duration at low amplitudes to control the risk of segregation<sup>[34]</sup>.

**Quality control for pervious pavements:** Properties of pervious concrete significantly depend on the porosity of hardened concrete, which is influenced by the compaction methodology.

To understand the mechanism of compaction, pervious concrete was cast using different techniques. The samples were tested for paste segregation and void distributions. Results show that on placement inside the mould, pervious concrete forms large cavities due to the interlocking of aggregates (Figure 16). Compression-based compaction fails to overcome the interlocking (Figure 16). Although vibration achieves better compaction, it also results in paste segregation and clogging of pores at the bottom surface. In comparison, mixed methods like proctor-based compaction yield better compaction without segregation. Since proctor-based compaction cannot be simulated in the field, the results of changing compaction can affect the field performance of pervious concrete. Therefore, it is recommended to follow compression-based compaction for the preparation of lab-scale specimens, to ensure replicability of results in the field.

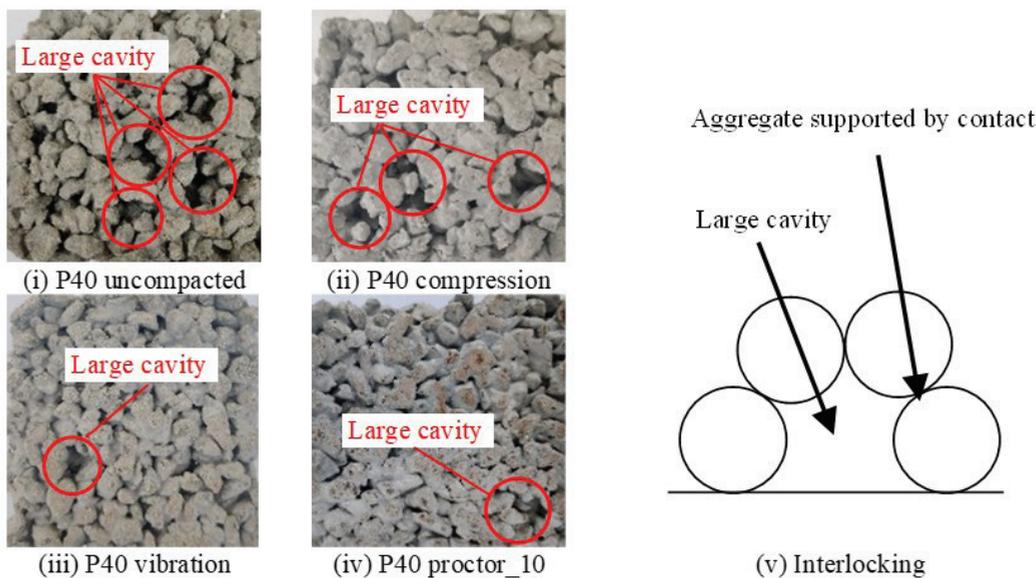


Figure 16: Formation of cavities during compaction of pervious concrete

*Functionally graded pavements:* Functionally graded concrete has been extensively investigated to improve the flexural performance of concrete. In a study on the functionally graded cubes, it was observed that the overall strength is higher than the strength of the weaker bottom layer<sup>[35]</sup>. Further investigation confirmed that the increased strength can be attributed to the stress distribution at the upper layer<sup>[35]</sup>. The results suggest that functionally graded pavements can be prepared with a slightly weaker bottom layer for better economy.

*Key contributions:* The various studies, at a meta-heuristic level, provide better techniques for the field application of concrete. At a fundamental level, the studies explore different variations in the mixing and casting procedures and observe their effect on the properties of concrete. A better understanding of the effects can help in improving the mixing and casting procedure of concrete.

*Limitations:* The replicability of the results significantly depends on the type of mix and instrument used. Therefore, the results present a direction to the stakeholders towards possible mixing and casting strategies for various concrete applications.

## 6. UNDERSTANDING FAILURE MECHANISMS AND DEVELOPMENT OF SAFETY GUIDELINES

The safe and robust industrial application of concrete while balancing economy is one of the major challenges for any stakeholder. Improving safety often involves taking a factor of safety, providing a buffer and/or introducing reserve strength to concrete. A conservative safety guideline increases costs, while a lenient safety guideline increases risks. Developing suitable safety guidelines often involves (i) understanding the failure mechanism, (ii) developing risk assessment models, and (iii) identifying safety guidelines.

### 6.1 Coupled effect of environmental exposures

In rigid pavements in cold climatic regions, concrete is subjected to fatigue and freeze-thaw. Exposure to both fatigue and freeze-thaw results in the creation and growth of micro-cracks till the concrete fails<sup>[36]</sup>. The two exposure phenomena act together in the field, while testing standards suggest an independent test for each exposure phenomenon to validate the durability of concrete in the lab<sup>[36]</sup>. One of the previous studies attempted to understand the coupled effect of exposure to fatigue and freeze-thaw by alternating applications of the two exposure phenomena<sup>[36]</sup>.

The study showed that a coupled exposure scenario creates a synergistic damage to concrete, based on the exposure pattern<sup>[36]</sup>. Alternating exposure scenario results in higher

compressive strength loss as compared to freeze-thaw, followed by fatigue cycles<sup>[36]</sup>. Furthermore, early exposure to freeze-thaw, the concrete showed higher residual compressive strength but lower flexural fatigue strength<sup>[36]</sup>. The results indicate that freeze-thaw before fatigue is more detrimental to flexural strength under fatigue, while fatigue before freeze-thaw is more detrimental to compressive strength<sup>[36]</sup>.

*Key contributions:* Through the specific scenario of fatigue and freeze-thaw, the study demonstrates the coupled effect of the exposure scenario.

*Limitations:* This work needs to be replicated over various scenarios to establish the exact mechanism of the coupled effect and translate it to safety guidelines. Similar studies are also needed to understand the interplay between various exposure scenarios.

### 6.2 Fire safety guidelines for limestone calcined clay cement

Limestone calcined clay cement (LC<sup>3</sup>) is a new type of ternary blended cement, and is considered an eco-friendlier alternative to ordinary Portland cement (OPC) and Portland pozzolana cement (PPC). One study investigated the thermal degradation patterns of hydrated LC<sup>3</sup> pastes and compared them with hydrated OPC and PPC pastes<sup>[37]</sup>. Higher thermal degradation was observed for LC<sup>3</sup> below 110 °C and across 600 °C – 750 °C, due to higher water content and calcium carbonates, respectively (Figure 17)<sup>[37]</sup>. At the same time, lower thermal degradation was observed for LC<sup>3</sup> across 125 °C – 155 °C and 400 °C – 460 °C, due to lower mono-sulfo-aluminate (Afm) and portlandite content (Figure 17)<sup>[37]</sup>. As part of the study, the loss in residual strength of LC<sup>3</sup> concrete is compared with OPC and PPC concrete<sup>[32]</sup>. The results confirmed a greater strength loss in LC<sup>3</sup> concrete when exposed to elevated temperatures, implying a higher risk<sup>[32]</sup>. LC<sup>3</sup> presented one case in which the introduction of a safety guideline can ensure a robust industrial application.

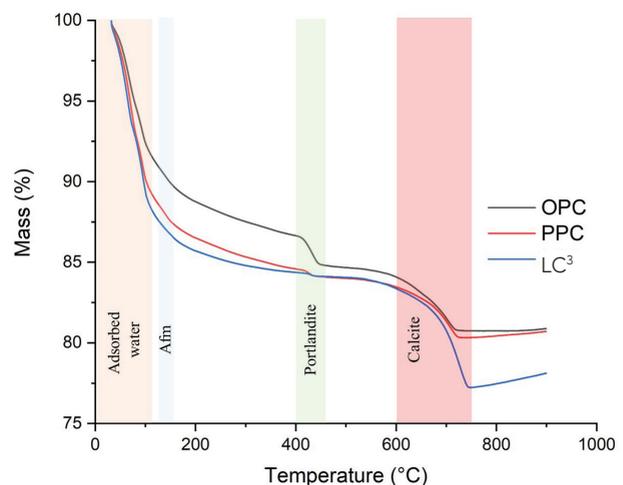


Figure 17: TGA curve for OPC, PPC, and LC<sup>3</sup> hydrated pastes

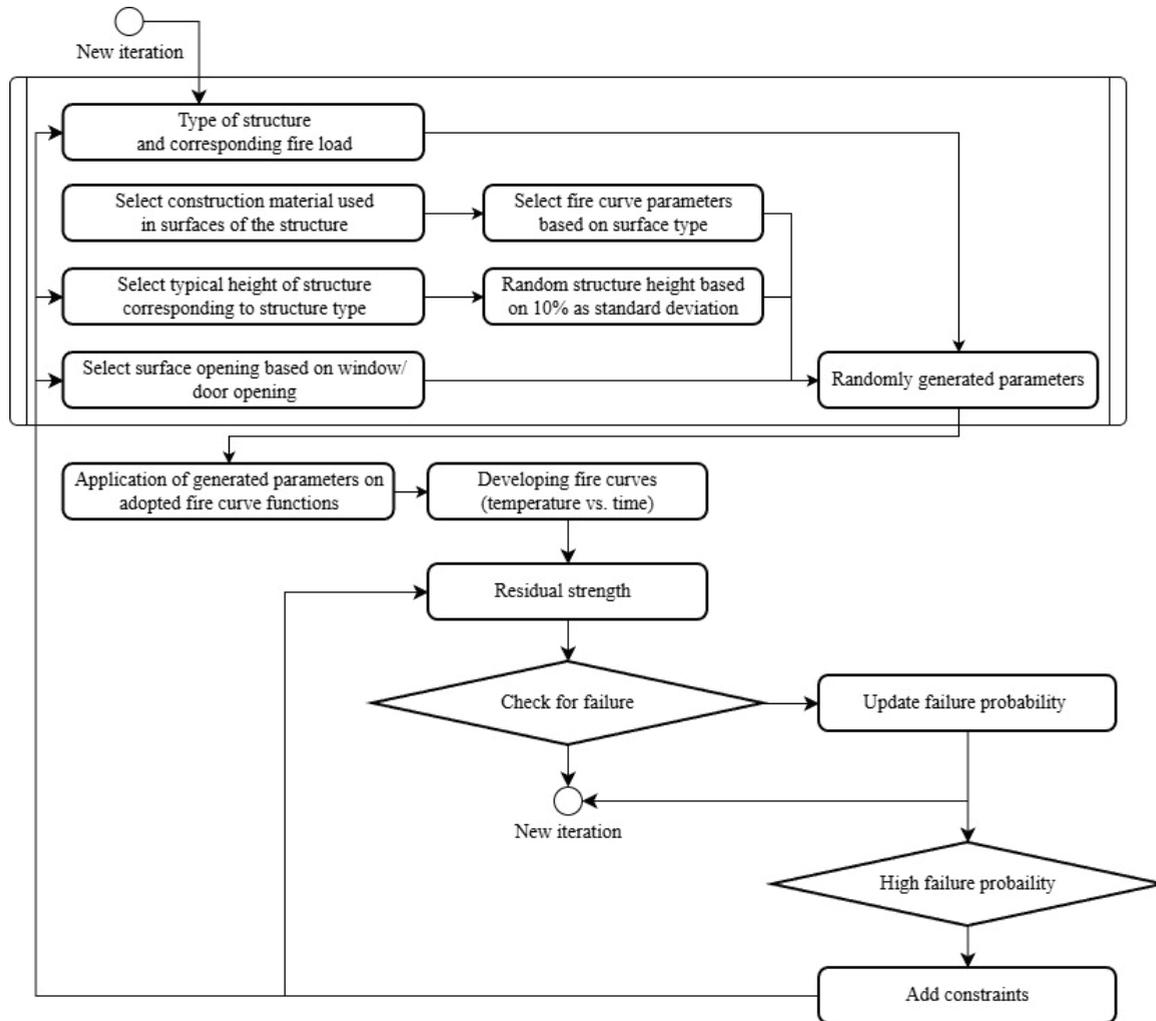


Figure 18: Outline of Monte Carlo simulation for fire risk assessment of concrete

In the case of a real fire, the resulting damage to concrete can vary with construction parameters. The study used different construction parameters that can be observed in the Indian construction industry to simulate millions of fire curves and identify the probability of damage to the LC<sup>3</sup> concrete<sup>[32]</sup>. Figure 18 shows the overview of the Monte Carlo simulation adopted in the study<sup>[32]</sup>. The advantage of determining failure probability is the development of safety guidelines while balancing economy. Concrete structures are designed with an acceptable risk or failure probability. Adding constraints to the Monte Carlo simulation can reduce the failure probability of concrete within acceptable margins. In the study on LC<sup>3</sup> concrete, constraints in the form of reserve strength and restricted structural application reduced the risks within acceptable margins<sup>[32]</sup>. The constraints represent the safety guidelines that can be extended to construction practices. Similar simulations can be used to determine risks and manage them to determine safety guidelines for robust industrial applications of alternative concrete under different failure mechanisms.

*Key contributions:* The study reiterates the application of Monte Carlo simulation for the development of risk assessment and exploits it for the development of safety guidelines. The concept of Monte Carlo for risk assessment is not novel. The present study, for the first time, extends the applicability of simulation by including actual parameters from the Indian construction industry and demonstrates the development of safety guidelines. The study also provides a better understanding towards the strength loss mechanism in LC<sup>3</sup> concrete, along with guidelines for fire safety.

*Limitations:* The simulation requires a complex framework and significant computational power. The study used a simplified framework by excluding reinforcement failure, thermal gradient, source of fire, and layout arrangement among various other factors. To compensate for the limitations of factors, the framework used a comparative analysis with OPC and LC<sup>3</sup>. Further expansion of the simulation is needed for true risk assessment, and development of a generalised framework applicable to a wide range of materials.

## 7. CONCLUDING REMARKS

The article presents several new observations in the field of concrete technology and the science behind them. The key contributions in concrete science can be summarised as follows:

- i. Characterisation-based classification using specific surface area and chemical composition presents a stronger correlation than source-based classification. This can be used for preliminary grouping of new and alternative SCMs.
- ii. Food waste as nutrient media, cow dung as foaming agent, thermo-mechanically treated recycled aggregates, and fine bone china coarse aggregates have demonstrated commercial viability at industrial scale applications. Waste tyre rubber and dimensional stone waste have demonstrated specialised applications as slip and fall-preventing concrete and naturally coloured geopolymers, respectively, providing direction towards new industrial applications.
- iii. *E. Coli DH5 $\alpha$*  favours strength gain over crack healing abilities. Further investigations with different bacteria can result in the development of bio-cement composites with different functional performance.
- iv. Surface irregularities in the abrasion drum improve the abrasive action and adhered mortar removal. The results can be used for improving the existing abrasive machines.
- v. Rheology of cement pastes can be split into reversible and irreversible changes. The proposed distinct shear rate protocol can maintain the cement pastes in a broken-down state, *i.e.*, without reversible changes, and isolate the irreversible changes in cement rheology.
- vi. Different mixing and compaction methods have demonstrated an influence on the final properties of the concrete. While some results can be adopted as they are, others can be used to improve industrial casting practices.
- vii. Coupled interaction of exposure scenarios has shown more serious damage to concrete. Further investigations are needed in the directions to improve failure mechanisms and improve the safety guidelines for the materials. Monte Carlo simulation presents one of the strategies for developing safety guidelines.

I hope that the discussion will support future innovations in concrete science and drive sustainable, robust technologies in the construction industry.

## 8. ACKNOWLEDGEMENT

The scientific autobiography showcases the contributions made by me, my research group, and other affiliates in the field of "concrete science". I want to acknowledge the fact

that, while this scientific autobiography may seem to credit the accomplishments to an individual, they have always been a team effort. While I may not be able to mention the effort of all the individuals who have helped me to this point, I wanted to take some words to acknowledge their part in this scientific autobiography.

## DATA AVAILABILITY STATEMENT

The relevant data sets will be made available on request.

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**Cite this article:** Chaudhary, S. (2026). "Impacting construction through concrete science focused on sustainable and robust translational research", *The Indian Concrete Journal*, Vol. 100, No. 1, pp. 66-82.